

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re application of :  
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Serial No. Not Yet Assigned :  
Filed Concurrently Herewith :

Pittsburgh, Pennsylvania  
May 14, 2001

PRELIMINARY AMENDMENT

Commissioner for Patents  
Washington, D.C. 20231

Sir:

Prior to initial examination, please amend the above-identified patent application as follows:

**IN THE SPECIFICATION:**

Please amend and delete section headings and amend specification paragraphs as follows. (Pursuant to 37 CFR 1.121, marked-up versions of the amended specification paragraphs are attached.)

On page 1, please amend the section heading "Filed of the invention" to read as follows:

Field of the invention

**Please delete the paragraph bridging pages 3 and 4 and insert the following replacement paragraph:**

Conventionally, as a method of purifying and removing metal ion impurities contained in an aqueous hydrogen peroxide solution, there is proposed a method comprising bringing a  $H^+$  type strongly acidic cation exchange resin containing a sulfonic acid group into contact with an aqueous hydrogen peroxide solution. However, merely by contacting the strongly acidic cation exchange resin with the aqueous hydrogen peroxide solution, although metal ion impurities such as Na are removed, it is difficult to remove impurities which are not dissolved completely in the aqueous hydrogen peroxide solution and/or which are originated from metal such as Al, Fe and Cr easily forming a complex with a hydroxide ion. There is a further problem that a cation exchange resin is deteriorated by contacting with the aqueous hydrogen peroxide solution, and thereby, a large amount of sulfate ion is generated from a  $SO_3H$  group of the cation exchange resin.

**On page 4, please delete the first complete paragraph and insert the following replacement paragraph:**

In an effort to solve the above problems, there is also known a method that after a contact of an aqueous hydrogen peroxide solution and a cation exchange resin, further a contact with an  $OH^-$  type strongly basic anion exchange resin having a quaternary ammonium group is carried out. By said method, the impurities which can not be removed by a strongly acidic cation exchange resin can be removed.

**On page 5, please delete the first complete paragraph and insert the following replacement paragraph:**

However, in the above cases, undissolved metal ion impurities in an aqueous hydrogen peroxide solution and metal ion impurities which easily form a complex together

with a hydroxide ion are not removed completely and remain. Therefore a high purity aqueous hydrogen peroxide can not be obtained. Due to the influence of the metal impurities which easily form a complex together with a hydroxide ion and the remaining metal ion impurities, it is difficult to fully prevent the decomposition of hydrogen peroxide. As a result, it is difficult to purify an aqueous hydrogen peroxide solution safely.

**On page 9, please delete the section heading "Object of the invention".**

**On page 9, please delete the first complete paragraph and insert the following replacement paragraph:**

An object of the present invention is to provide a purifying process of an aqueous hydrogen peroxide solution in which metal ion impurities and the metal ion impurities are removed as completely as possible.

**On page 10, please delete the second complete paragraph and insert the following replacement paragraph:**

As described above, the treatments of three-step or four-step of ion exchange resin lead to the production of a high-purity aqueous hydrogen peroxide solution from which ion impurities are removed as completely as possible.

**On page 12, please delete the first complete paragraph and insert the following replacement paragraph:**

Said purified aqueous hydrogen peroxide solution is preferably obtained by filtrating solid impurities contained in the aqueous hydrogen peroxide solution to which a flocculating agent has been preliminarily added, by a fine filter. By preliminarily adding a flocculating agent into an aqueous hydrogen peroxide solution and filtrating impurities by a fine filter, the insoluble metal ion impurity components which can not be removed by the ion exchange treatment are removed. As a result, metal ion impurities in the aqueous hydrogen peroxide solution can be removed up to a ppt level ( $1/10^{12}$ ) or its vicinities. Further, such

filtration leads high duplicativity of the metal ion impurities removing level which is accomplished at a treatment by an ion exchange resin, as described later.

**Please delete the paragraph bridging pages 18 and 19 and insert the following replacement paragraph:**

The  $H^+$  type cation exchange resin used for the invention is one conventionally known as a strongly acidic cation exchange resin. Generally, the strongly acidic cation exchange resin preferably has a network structure in which a sulphonic acid group is introduced into a styrene-divinylbenzene crosslinked copolymer. The degree of cross linkage of such cation exchange resin is usually 6 to 10, preferably 7 to 9.

**On page 19, please delete the first complete paragraph and insert the following replacement paragraph:**

The  $H^+$  type strongly acidic cation exchange resin includes, for example, PK216, SK1B and IR-120B.

**On page 19, please delete the second complete paragraph and insert the following replacement paragraph:**

The  $H^+$  type cation exchange resin is preferably regenerated by repeating a process, 2 times or more, preferably 2 to 12 times, in which a cation exchange resin is treated with a downflow of inorganic acid aqueous solution (a regenerant) and then washed with upflow of ultra-pure water.

**Please delete the paragraph bridging pages 19 and 20 and insert the following replacement paragraph:**

Usually, regeneration of a cation exchange resin is carried out by a process that the regenerant aqueous solution is passed through and then ultra-pure water is passed through for washing the resin. In the present invention, a cycle of regenerant flowing/ultra-pure water washing is desirably repeated particularly 2 or more times. By repeating the

inorganic acid aqueous solution/ultra-pure water flow, the exchange resin can be effectively and homogeneously regenerated and further can be washed up to the inside of the resin due to contraction and swelling of the resin.

**On page 24, please delete the first complete paragraph and insert the following replacement paragraph:**

To convert into a hydroxide ion type anion exchange resin, it is preferred that a process, in which an anion exchange resin is treated with a downflow of strongly alkali aqueous solution (regenerant) and then treated with upflow of ultra-pure water, is repeated 2 or more times thereby to regenerate. Usually, an anion exchange resin is brought into contact with a regenerant aqueous solution by a process that the regenerant aqueous solution is passed through and washed by ultra-pure water. In the present invention, a cycle of passing of regenerant/washing with ultra-pure water is desirably repeated particularly 2 or more times. By repeating the strongly alkali aqueous solution/ultra-pure water flow, the exchange resin can be effectively and homogeneously regenerated and further can be washed to the inside of the resin due to contraction and swelling of the resin.

**On page 26, please delete the second complete paragraph and insert the following replacement paragraph:**

As the carbonate or bicarbonate, a conventionally known carbonate or bicarbonate such as sodium carbonate, sodium bicarbonate, potassium carbonate and potassium bicarbonate is used. By repeating the carbonate or bicarbonate aqueous solution/ultra-pure water flow, as described above, the exchange resin can be effectively and homogeneously regenerated and further can be washed to the inside of the resin due to contraction and swelling of the resin.

**On page 31, please delete the second complete paragraph and insert the following replacement paragraph:**

By repeating the fluorine compound aqueous solution/ultra-pure water flow, the ion exchange resin can be effectively and homogeneously regenerated and further can be washed up to the inside of the resin due to contraction and swelling of the resin.

**Please delete the paragraph bridging pages 35 and 36 and insert the following replacement paragraph:**

After the phosphorus based compound is added, it is preferred that the aqueous hydrogen peroxide solution has been aged for usually more than a day, preferably 1 to 5 days. Aging may be carried out with or without stirring. By the aging, insoluble metal ion impurities in the aqueous hydrogen peroxide solution are flocculated and grown to be filtrated.

**On page 37, please delete the section heading "Effect of the invention".**

**IN THE CLAIMS:**

**Please cancel the previous versions of claims 3-9 and 12 and insert the amended versions of claims 3-9 and 12 as follows. (Pursuant to 37 CFR 1.121, marked-up versions of these claims are attached.)**

3. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 1, wherein the aqueous hydrogen peroxide solution is contacted with an adsorption resin before contacted with the  $H^+$  type cation exchange resin.

4. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 1, wherein said  $H^+$  type cation exchange resin is regenerated by repeating a process, two or more times, in which the cation exchange resin is treated with a downward flowing inorganic acid aqueous solution and then washed with ultra-pure water.

5. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 1, wherein the carbonate ion ( $\text{CO}_3^{2-}$ ) type or bicarbonate ion ( $\text{HCO}_3^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with a sodium carbonate or sodium bicarbonate aqueous solution and then washed with ultra-pure water.

6. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 2, wherein the fluoride ion ( $\text{F}^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with at least one fluorine compound aqueous solution selected from the group consisting of sodium fluoride, potassium fluoride and ammonium fluoride and then washed with ultra-pure water.

7. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 3, wherein the adsorption resin is regenerated by treating with an alcohol aqueous solution as a regenerant and then washing with ultra-pure water.

8. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 1, wherein the hydrogen peroxide concentration in the aqueous hydrogen peroxide solution is 40 to 70 % by weight.

9. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 1, wherein said purified aqueous hydrogen peroxide

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solution is obtained by filtrating a solid impurities contained in the aqueous hydrogen peroxide solution to which a flocculating agent has been preliminarily added, by a fine filter.

12. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 9, wherein the fine filter has an average pore size of 0.2  $\mu\text{m}$  or less.

**Please add new claims 13-26 as follows:**

13. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 2, wherein the aqueous hydrogen peroxide solution is contacted with an adsorption resin before contacted with the  $\text{H}^+$  type cation exchange resin.

14. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 2, wherein said  $\text{H}^+$  type cation exchange resin is regenerated by repeating a process, two or more times, in which the cation exchange resin is treated with a downward flowing inorganic acid aqueous solution and then washed with ultra-pure water.

15. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 3, wherein said  $\text{H}^+$  type cation exchange resin is regenerated by repeating a process, two or more times, in which the cation exchange resin is treated with a downward flowing inorganic acid aqueous solution and then washed with ultra-pure water.

16. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 2, wherein the carbonate ion ( $\text{CO}_3^{2-}$ ) type or bicarbonate ion ( $\text{HCO}_3^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with a sodium carbonate or sodium bicarbonate aqueous solution and then washed with ultra-pure water.



17. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 3, wherein the carbonate ion ( $\text{CO}_3^{2-}$ ) type or bicarbonate ion ( $\text{HCO}_3^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with a sodium carbonate or sodium bicarbonate aqueous solution and then washed with ultra-pure water.

18. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 4, wherein the carbonate ion ( $\text{CO}_3^{2-}$ ) type or bicarbonate ion ( $\text{HCO}_3^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with a sodium carbonate or sodium bicarbonate aqueous solution and then washed with ultra-pure water.

19. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 3, wherein the fluoride ion ( $\text{F}^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with at least one fluorine compound aqueous solution selected from the group consisting of sodium fluoride, potassium fluoride and ammonium fluoride and then washed with ultra-pure water.

20. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 4, wherein the fluoride ion ( $\text{F}^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is treated with at least one fluorine compound aqueous solution selected from the group consisting of sodium fluoride, potassium fluoride and ammonium fluoride and then washed with ultra-pure water.

21. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 5, wherein the fluoride ion ( $\text{F}^-$ ) type anion exchange resin is regenerated by repeating a process, two or more times, in which the anion exchange resin is

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treated with at least one fluorine compound aqueous solution selected from the group consisting of sodium fluoride, potassium fluoride and ammonium fluoride and then washed with ultra-pure water.

22. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 4, wherein the adsorption resin is regenerated by treating with an alcohol aqueous solution as a regenerant and then washing with ultra-pure water.

23. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 5, wherein the adsorption resin is regenerated by treating with an alcohol aqueous solution as a regenerant and then washing with ultra-pure water.

24. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 6, wherein the adsorption resin is regenerated by treating with an alcohol aqueous solution as a regenerant and then washing with ultra-pure water.

25. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 10, wherein the fine filter has an average pore size of 0.2  $\mu\text{m}$  or less.

26. The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 11, wherein the fine filter has an average pore size of 0.2  $\mu\text{m}$  or less.

REMARKS

The specification has been amended to place the application in conformance with standard United States patent practice.

Claims 3-9 have been amended to bring the claims into conformance with standard United States patent practice.

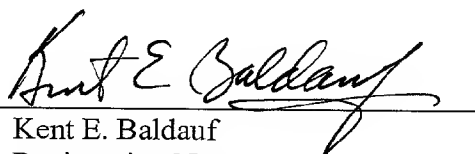
New claims 13-26 have been added to further define the invention.

Examination and allowance of pending claims 1-26 are respectfully requested.

Respectfully submitted,

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## MARKED-UP AMENDED SPECIFICATION HEADING AND PARAGRAPHS

### Page 1, first section heading

[Filed of the invention]

Field of the invention

### Paragraph bridging pages 3 and 4

Conventionally, as a method of purifying and removing metal ion impurities contained in an aqueous hydrogen peroxide solution, there is proposed a method [comprises] comprising bringing a  $H^+$  type strongly acidic cation exchange resin containing a sulfonic acid group into contact with an aqueous hydrogen peroxide solution. However, merely by contacting the strongly [acid] acidic cation exchange resin with the aqueous hydrogen peroxide solution, although metal ion impurities such as Na are removed, it is difficult to remove impurities which are not dissolved completely in the aqueous hydrogen peroxide solution and/or which are originated from metal such as Al, Fe and Cr easily forming a complex with a hydroxide ion. There is a further problem that a cation exchange resin is deteriorated by contacting with the aqueous hydrogen peroxide solution, and thereby, a large amount of sulfate ion is generated from a  $SO_3H$  group of the cation exchange resin.

### Page 4, first complete paragraph

[Due] In an effort to solve the above problems, there is also known a method that after a contact of an aqueous hydrogen peroxide solution and a cation exchange resin, further a contact with an  $OH^-$  type strongly basic anion exchange resin having a [quarternary] quaternary ammonium group is carried out. By said method, the impurities which can not be removed by a strongly acidic cation exchange resin can be removed.

**Page 5, first complete paragraph**

However, in the above cases, [not dissolved] undissolved metal ion impurities in an aqueous hydrogen peroxide solution and metal ion impurities [easy to] which easily form a complex together with a hydroxide ion are not removed completely and remain. Therefore a high purity aqueous hydrogen peroxide can not be obtained. Due to the influence of the metal impurities [easy to] which easily form a complex together with a hydroxide ion and the [remained] remaining metal ion impurities, it is difficult to fully prevent the decomposition of hydrogen peroxide. As a result, it is difficult to purify an aqueous hydrogen peroxide solution safely.

**Page 9, first complete paragraph**

[The present invention has an] An object of the present invention is to provide a purifying process of an aqueous hydrogen peroxide solution in which metal ion impurities and the metal ion impurities are removed as completely as possible.

**Page 10, second complete paragraph**

As described above, the treatments of three-step or four-step of ion exchange resin lead to [produce] the production of a high-purity aqueous hydrogen peroxide solution from which ion impurities are removed as completely as possible.

**Page 12, first complete paragraph**

Said purified aqueous hydrogen peroxide solution is preferably obtained by filtrating [a] solid impurities contained in the aqueous hydrogen peroxide solution to which a flocculating agent has been [preliminary] preliminarily added, by a fine filter. By [preliminary] preliminarily adding a flocculating agent into an aqueous hydrogen peroxide solution and filtrating impurities by a fine filter, the insoluble metal ion impurity components which can not be removed by the ion exchange treatment are removed. As a result, metal ion impurities in the aqueous

hydrogen peroxide solution can be removed up to a ppt level ( $1/10^{12}$ ) or its vicinities. Further, such filtration leads high duplicativity of the metal ion impurities removing level which is accomplished at a treatment by an ion exchange resin, as described later.

**Paragraph bridging pages 18 and 19**

The  $H^+$  type cation exchange resin used for the invention is one conventionally known as a strongly [acid] acidic cation exchange resin. Generally, the strongly [acid] acidic cation exchange resin preferably has a network structure in which a sulphonic acid group is introduced into a styrene-divinylbenzene crosslinked copolymer. The degree of cross linkage of such cation exchange resin is usually 6 to 10, preferably 7 to 9.

**Page 19, first complete paragraph**

The  $H^+$  type strongly [acid] acidic cation exchange resin includes, for example, PK216, SK1B and IR-120B.

**Page 19, second complete paragraph**

The  $H^+$  type cation exchange resin is preferably regenerated by repeating a process, 2 times or more, preferably 2 to 12 times, in which [an] a cation exchange resin is treated with a downflow of inorganic acid aqueous solution (a regenerant) and then washed with upflow of ultra-pure water.

**Paragraph bridging pages 19 and 20**

Usually, regeneration of a cation exchange resin is carried out by a process that the regenerant aqueous solution is passed through and then ultra-pure water is passed through for washing the resin. In the present invention, a cycle of regenerant flowing/ultra-pure water washing is desirably repeated particularly 2 or more times. By repeating the inorganic acid aqueous solution/ultra-pure water flow,

the exchange resin can be effectively and [homogenously] homogeneously regenerated and further can be washed up to the inside of the resin due to contraction and swelling of the resin.

**Page 24, first complete paragraph**

To convert into a hydroxide ion type anion exchange resin, it is preferred that a process, in which an anion exchange resin is treated with a downflow of strongly alkali aqueous solution (regenerant) and then treated with upflow of ultra-pure water, is repeated 2 or more times thereby to regenerate. Usually, an anion exchange resin is brought into contact with a regenerant aqueous solution by a process that the regenerant aqueous solution is passed through and washed by ultra-pure water. In the present invention, a cycle of passing of regenerant/washing with ultra-pure water is desirably repeated particularly 2 or more times. By repeating the strongly alkali aqueous solution/ultra-pure water flow, the exchange resin can be effectively and [homogenously] homogeneously regenerated and further can be washed to the inside of the resin due to contraction and swelling of the resin.

**Page 26, second complete paragraph**

As the carbonate or bicarbonate, a conventionally known carbonate or bicarbonate such as sodium carbonate, sodium bicarbonate, potassium carbonate and potassium bicarbonate is used. By repeating the carbonate or bicarbonate aqueous solution/ultra-pure water flow, as described above, the exchange resin can be effectively and [homogenously] homogeneously regenerated and further can be washed to the inside of the resin due to contraction and swelling of the resin.

**Page 31, second complete paragraph**

By repeating the fluorine compound aqueous solution/ultra-pure water flow, the ion exchange resin can be effectively and [homogenously] homogeneously

regenerated and further can be washed up to the inside of the resin due to contraction and swelling of the resin.

**Paragraph bridging pages 35 and 36**

After the phosphorus based compound is added, it is preferred that the aqueous hydrogen peroxide solution has been aged for usually more than a day, preferably 1 to 5 days. Aging may be carried out with or without stirring. By the aging, insoluble metal ion impurities in the aqueous hydrogen peroxide solution are [floccurated] flocculated and grown to be filtrated.



**MARKED-UP AMENDED CLAIMS**

3. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in claim 1 [or 2], wherein the aqueous hydrogen peroxide solution is contacted with an adsorption resin before contacted with the  $H^+$  type cation exchange resin.

4. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 1 [to 3], wherein said  $H^+$  type cation exchange resin is regenerated by repeating a process, [2] two or more times, in which the cation exchange resin is treated with a downward flowing inorganic acid aqueous solution and then washed with ultra-pure water.

5. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 1 [to 4], wherein the carbonate ion ( $CO_3^{2-}$ ) type or bicarbonate ion ( $HCO_3^-$ ) type anion exchange resin is regenerated by repeating a process, [2] two or more times, in which the anion exchange resin is treated with a sodium carbonate or sodium bicarbonate aqueous solution and then washed with ultra-pure water.

6. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 2 [to 5], wherein the fluoride ion ( $F^-$ ) type anion exchange resin is regenerated by repeating a process, [2] two or more times, in which the anion exchange resin is treated with at least one fluorine compound aqueous solution selected from the group consisting of sodium

fluoride, potassium fluoride and ammonium fluoride and then washed with ultra-pure water.

7. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 3 [to 6], wherein the adsorption resin is regenerated by treating with an alcohol aqueous solution as a regenerant and then washing with ultra-pure water.

8. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 1 [to 7], wherein the hydrogen peroxide concentration in the aqueous hydrogen peroxide solution is 40 to 70 % by weight.

9. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 1 [to 8], wherein said purified aqueous hydrogen peroxide solution is obtained by filtrating a solid impurities contained in the aqueous hydrogen peroxide solution to which a flocculating agent has been [preliminary] preliminarily added, by a fine filter.

12. (Amended) The process for producing a purified aqueous hydrogen peroxide solution as claimed in [any one of claims] claim 9 [to 11], wherein the fine filter has an average pore size of 0.2  $\mu\text{m}$  or less.